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Mixed-Ligand Complexes of Ni(II), Cu(II), and Zn(II) with 5-Chlorosalicylaldehyde and β-Diketones

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ABSTRACT

Mixed-ligand complexes of the types M(5-Clsal)L and Ni(5-Clsal)L(H₂O)₂ (where M = copper(II) or zinc(II), 5-ClsalH = 5-chlorosalicylaldehyde and HL = 2,4-pentanedione, 1-phenyl-1,3-butanedione, or 1,3-diphenyl-1,3-propanedione) have been synthesized by 1:1:1 molar reactions of metal chlorides with 5-chlorosalicylaldehyde and β -diketones. The resulting complexes have been characterized by elemental analyses, TLC, conductances, thermal analysis, magnetic moments, IR, electronic, and ¹H NMR spectra.

Key Words: Mixed-ligand complexes; Thin layer chromatography; Magnetic moments; IR spectra; Electronic spectra.

943

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INTRODUCTION

944

Mixed-ligand complexes play important roles in biological processes, as exemplified by many instances in which enzymes are known to be activated by metal ions.^[1,2] Such complexes have been implicated in the storage and transport of active substances through membranes.^[3] Interest in mixed chelation also arises from the study of mechanisms of organic reactions, where the metal ion or the bound ligand is activated for selective reactions. Hydrolysis of α -amino acid esters, stereoselective decarboxylation, Schiff base formation, and amino acid oxidase models provide typical examples.^[4] Many mixed-ligand complexes are finding applications in the microelectronic industry, chemical vapor deposition of metals and as drugs.^[5,6] Hence, the investigations on mixed-ligand complexes have been carried out in our laboratories. These investigations will help in understanding the role of metal ions in the active sites of metalloenzymes, studying the toxic effects of metal ions, detoxification mechanisms, and drug designing.^[7]

Mixed-ligand complexes of Ni(II), Cu(II), and Zn(II) of the type MLL' with aldehydes and ketones have been reported.^[8–10] However, the mixedligand complexes of these metals with substituted salicylaldehydes have not been reported, so far except for complexes of alkaline earth metals reported earlier from our laboratories.^[11–13] In the present paper, mixedligand complexes of the types M(5-Clsal)L (M = Cu(II) and Zn(II)) and Ni(5-Clsal)L(H₂O)₂ (where 5-ClsalH = 5-chlorosalicylaldehyde and HL = 2,4-pentanedione (acacH), 1-phenyl-1,3-butanedione (bzacH), or 1,3-diphenyl-1,3-propanedione (dbzmH), Fig. 1) are described.





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EXPERIMENTAL

Materials

5-Chlorosalicylaldehyde (Aldrich), benzoylacetone (Fluka), and dibenzoylmethane (Sisco) were purified by recrystallization from hot ethanol. Acetylacetone (K. Light) and ethanol were purified by distillation. NiCl₂ · 6H₂O (Fluka), CuCl₂ · 2H₂O (Fluka), and ZnCl₂ (Fluka) were of AR grade.

Synthesis of Mixed-Ligand Complexes of Ni(II), Cu(II), and Zn(II)

To an ethanolic solution of Ni(II) or Cu(II) chloride (about 3.4 mmol in 10 mL) a mixture of the two-carbonyl compounds (3.4 mmol each) in ethanol (10 mL) was added with stirring. A clear solution was obtained. The pH of the reaction mixture was raised to \sim 5.5 by adding 5% aqueous sodium hydroxide solution dropwise with stirring. The pH was measured with the help of pH paper and stirring was continued for 3-4 hr. The solid complexes, which separated were filtered off, washed with ethanol, and dried under reduced pressure.

The zinc(II) mixed-ligand complexes were prepared similarly, except a 5% aqueous ammonia solution was used instead of a 5% aqueous sodium hydroxide solution.

Analytical Methods

Nickel and zinc were determined volumetrically by EDTA titration using eriochrome black T as indicator. Copper was determined volumetrically by sodium thiosulfate solution using starch as indicator.^[14] Carbon and hydrogen analyses were carried out on a Heraeus Carlo Erba 1108 instrument. Specific conductances were measured at room temperature in DMSO by a Systronics direct reading 304 conductivity meter, using a glass cell having a cell constant of 1.0 cm⁻¹. TG and differential thermal analyses (DTA) studies were carried out on a Rigaku Thermoflex PTC-10A instrument under nitrogen atmosphere. Infrared spectra of KBr pellets of the complexes were recorded in the region 400-4000 cm⁻¹ on a Nicolet Magna-550 FT IR spectrophotometer. Electronic spectra were recorded in DMSO on a Hitachi U-2000 spectrophotometer in the range 200-1100 nm. ¹H NMR spectra were recorded in DMSO-d₆ on a Jeol FX 90 Q FT NMR spectrometer at 90 MHz using TMS as reference. Magnetic measurements were carried out at room temperature on a Gouy balance using Hg[Co(CNS)₄] as calibrant.

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RESULTS AND DISCUSSION

946

The reactions of the hydrated metal ions with 5-chlorosalicylaldehyde and β -diketones (acetylacetone, benzoylacetone, or dibezoylmethane) in 1:1:1 molar ratios result in the formation of mixed-ligand complexes [(I) or (II) in Fig. 2].

The Ni(II) complexes are green, the Cu(II) complexes are grey, and the Zn(II) complexes are yellow solids. They are insoluble in chloroform, carbon tetrachloride, benzene, nitrobenzene, and methanol but soluble in DMSO. The properties and analyses of the complexes are recorded in Table 1. The conductances of the complexes are very low $(5-30 \,\Omega^{-1} \,\mathrm{cm}^2 \,\mathrm{mol}^{-1})$ indicating their non-electrolytic nature.^[15]

Thin Layer Chromatography

TLC of all the mixed-ligand complexes was performed on silica gel G using benzene-petroleum ether (b.p. 40-60 °C) (1:1, v/v) as the mobile



Figure 2. Synthesis of mixed-ligand complexes.

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	lable 1. Analyses	and characteristics of	the mixed	d-ligand co	mplexes o	I NI(II), C	u(11), and Zn(11).	
	-	Color and		Analyses	, % found	(calcd.)	Molar	
S. no.	Complex, emp. rormula, formula wt.	temp. (°C)	r ieid (%)	Metal	С	Н	$(\Omega^{-1} \mathrm{cm}^2 \mathrm{mol}^{-1})$	$\mu_{\rm eff}$ (B.M.)
(1)	[Ni(5-Clsal)(acac)(H_2O) ₂],	Green, 230–235	52	16.73	40.75	4.12	18	2.73
	$C_{12}H_{15}O_6CINi$, 349.14			(16.80)	(41.24)	(4.30)		
(5)	$[Ni(5-Clsal)(bzac)(H_2O)_2],$	Green, 270–275	49	14.38	48.85	4.30	13	2.87
	$C_{17}H_{17}O_6CINi$, 411.14			(14.27)	(49.62)	(4.13)		
(3)	$[Ni(5-Clsal)(dbzm)(H_2O)_2],$	Green, 180–185	85	12.30	54.93	3.87	27	2.84
	C ₂₂ H ₁₉ O ₆ CINi, 473.14			(12.40)	(55.80)	(4.02)		
(4)	[Cu(5-Clsal)(acac)],	Grey, 188–193	76	19.85	44.30	3.35	9	1.76
	$C_{12}H_{11}O_4CICu$, 318.00			(19.98)	(45.28)	(3.46)		
(2)	[Cu(5-Clsal)(bzac)],	Grey, 188–193	91	16.84	53.44	3.55	L	2.18
	$C_{17}H_{13}O_4CICu$, 380.00			(16.72)	(53.68)	(3.42)		
9	[Cu(5-Clsal)(dbzm)],	Grey, 195–200	92	14.48	60.53	3.49	5	1.96
	C ₂₂ H ₁₅ O ₄ ClCu, 442.00			(14.37)	(59.72)	(3.39)		
6	[Zn (5-Clsal)(acac)],	Yellow, 210–215	41	20.53	44.20	3.50	26	Diamagnetic
	$C_{12}H_{11}O_4CIZn$, 319.83			(20.44)	(45.02)	(3.44)		
(8)	[Zn(5-Clsal)(bzac)],	Yellow, 190–195	43	17.25	52.17	3.53	30	Diamagnetic
	$C_{17}H_{13}O_4CIZn$, 381.83			(17.12)	(53.43)	(3.40)		
6)	[Zn(5-Clsal)(dbzm)],	Yellow, 239–244	44	14.82	58.64	3.48	15	Diamagnetic
	C ₂₂ H ₁₅ O ₄ ClZn, 443.83			(14.73)	(59.48)	(3.38)		



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phase and the retention times were compared with those of the corresponding bis-complexes, e.g., $M(5-Clsal)_2$ and ML_2 . All the mixed-ligand complexes show single spots with R_f values being intermediate between the two corresponding symmetrical bis-complexes. This indicates that these are indeed mixed-ligand complexes rather than mixtures of the two corresponding bis-complexes.

948

Thermal Analysis

Thermogravimetric (TGA) and DTA of the mixed-ligand complexes were carried out to confirm the presence of coordinated water molecules. The weight loss observed by TGA in the 120-150 °C range for the Ni(II) complexes corresponds to two coordinated water molecules, e.g., in Ni(5-Clsal)(acac) (H₂O)₂, the weight loss found is 10.1%, calcd. 10.3%. This is confirmed by a sharp endothermic peak observed in the 130-175 °C range in the DTA of the nickel(II) complexes. TGA and DTA studies of the Cu(II) and Zn(II) complexes confirm the absence of coordinated water molecules in these complexes.

Infrared Spectra

In the IR spectra of the mixed-ligand complexes, strong absorption bands in the region $1580-1650 \text{ cm}^{-1}$ maybe attributed to coordinated C=O groups and those in the region $1475-1580 \text{ cm}^{-1}$ to C=C stretching modes. In free acetylacetone and benzoylacetone, the ν (C=O) band^[16] has been reported at 1724 cm^{-1} and in 5-chlorosalicylaldehyde, the ν (C=O) band appears at 1680 cm^{-1} . Thus, the shift of ν (C=O) bands to the lower wave number side in the mixed-ligand complexes supports the coordination of the C=O group to the metal ion. Shifting of these bands to lower wave number on complexation with the metal ion has been reported.^[17,18] In the spectra of the mixed-ligand complexes, weak to medium intensity absorption bands in the region $405-525 \text{ cm}^{-1}$, which are not present in the free ligands, maybe attributed to ν (M–O) vibrations.^[19] However, the IR data could be as well explained by [M(5-Clsal)₂ + ML₂] mixture as by M(5-Clsal)L.

Magnetic Moments

For the Cu(II) complexes, the μ_{eff} values (at ~302 K) are in the range 1.76–2.18 B.M., which are slightly higher than those expected for a d⁹ system



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(1.73 B.M.) and maybe due to the incomplete quenching of orbital contribution to the magnetic moment^[20] or due to spin–orbit coupling.^[21] The $\mu_{\rm eff}$ values in the range 2.00–2.25 B.M. have been reported for mixed-ligand complexes of Cu(II) with 3-chloro-, 3-nitro-, or aryl-substituted 2,4-pentane-diones and 2,2'-bipyridine or 1,10-phenanthroline.^[22] For the Ni(II) complexes, the $\mu_{\rm eff}$ values were observed in the range 2.72–2.87 B.M. suggesting a distorted octahedral geometry.^[23] The Zn(II) complexes are diamagnetic.

Electronic Spectra

The electronic spectra of the Cu(II) complexes exhibit a broad band at 655–665 nm ($\varepsilon = 78-107 \,\mathrm{L}\,\mathrm{mol}^{-1}\,\mathrm{cm}^{-1}$) suggesting a square-planar geometry.^[24] This broad band does not show any sign of splitting, suggesting that all the d–d transitions are lying within this broad band.^[25] In the case of the nickel(II) complexes, the absorption band at 694–715 nm ($\varepsilon = 12-17 \,\mathrm{L}\,\mathrm{mol}^{-1}\,\mathrm{cm}^{-1}$) maybe assigned to the ${}^{3}A_{2g}(F) \rightarrow {}^{3}T_{1g}(F)$ transition.^[26] The ${}^{3}A_{2g}(F) \rightarrow {}^{3}T_{1g}(F)$ transition band merges with the strong charge transfer band at 437–443 nm ($\varepsilon = 318-615 \,\mathrm{L}\,\mathrm{mol}^{-1}\,\mathrm{cm}^{-1}$). These observed transitions suggest a pseudo-octahedral environment around the nickel atom in the mixed-ligand complexes.^[26] Similar results have been reported for octahedral nickel(II) mixed-ligand complexes with β -diketones and nitrogen donor ligands.^[27] In all the mixed-ligand complexes of Ni(II), Cu(II), and Zn(II), strong absorption bands at 311–325 nm ($\varepsilon = 320-547 \,\mathrm{L}\,\mathrm{mol}^{-1}\,\mathrm{cm}^{-1}$) and 367–372 nm ($\varepsilon = 273-533 \,\mathrm{L}\,\mathrm{mol}^{-1}\,\mathrm{cm}^{-1}$) maybe due to intra-ligand transitions ($n \rightarrow \pi^*$ and $\pi \rightarrow \pi^*$) and the band at 399–443 nm ($\varepsilon = 318-615 \,\mathrm{L}\,\mathrm{mol}^{-1}\,\mathrm{cm}^{-1}$) maybe a charge transfer band.

Nuclear Magnetic Resonance Spectra

In free 5-chlorosalicylaldehyde the CH proton gives a singlet at δ 10.36 ppm, which is shifted upfield in the mixed-ligand complexes of Zn(II) and then appears at δ 10.01–10.07 ppm confirming the coordination of the C=O group of the aldehyde to the zinc atom. The OH proton peak of free 5-chlorosalicylaldehyde (δ 10.88 ppm) disappears in the spectra of the mixed-ligand complexes supporting the binding of the zinc atom to the phenolic oxygen of the OH group through the replacement of the hydrogen atom. In free acetylacetone, benzoylacetone, and dibenzoylmethane, the singlets due to the CH protons have been reported at δ 5.54, 6.18, and 6.82 ppm, respectively.^[28] In the Zn(II) mixed-ligand complexes, these

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singlets are observed at δ 5.44, 5.98, and 6.71 ppm, respectively. In free acetylacetone and benzoylacetone, the CH₃ proton singlets have been reported at δ 1.99 and 2.19 ppm, respectively,^[28] and in the Zn(II) mixed-ligand complexes, these singlets appear at δ 1.77 and 2.01 ppm, respectively. Thus, the upfield shift of the CH and CH₃ protons of the β -diketone ligands in the mixed-ligand complexes confirms the coordination of these ligands to the zinc atom. Similar upfield shifts of CH and CH₃ protons have been reported for β -diketonates of Group II, III, and IV metals.^[28–30]

Thus, the spectral and magnetic studies support the proposed structures of the mixed-ligand complexes in Fig. 2.

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